# METHOD AND EQUIPMENT FOR LIQUID-LIQUID EXTRACTION

The invention relates to a method for reversing the dispersion formed in the mixing section of liquid-liquid extraction and kept condensed in the separation section and the separated solutions from the rear end of the separation section to flow back towards the feed end of the separation section as two separate streams. The invention also refers to the extraction equipment for implementing the reversed flow.

The method and equipment relate in particular to an extraction process used in the recovery of metals. Extraction facilities recovering valuable metals such as copper, uranium, cobalt, nickel, zinc and molybdenum fall into this category. In all these extraction processes, a valuable metal-containing aqueous solution is brought into contact with an organic solution in the mixing section of extraction. Thus a dispersion of two solutions that are insoluble in each other is formed. The solutions in the dispersion are separated from each other into two successive layers in the separation section of extraction with an ever-decreasing dispersion band between the separating layers. During the mixing stage at least one of the valuable metals in the aqueous solution is transferred to the organic phase, from which the valuable metal is recovered by stripping. Extraction is performed in an arrangement of equipment, where the mixing and settling sections are either located one on top of the other (column) or horizontally on more or less the same level. Almost always in cases when large-scale extraction of weak solutions is concerned, such as copper extraction, the equipment is positioned in a substantially horizontal position. When we refer to extraction hereinafter, we are referring substantially to equipment positioned on the same level.

The recovery of metals often requires many mixing-separation units or mixer-settlers, which are usually connected to each other on the countercurrent principle. The number of extraction steps varies greatly depending on the

process and may be between 2 and 20. For example in copper extraction there are usually around 4 – 6 steps. So far units have almost always been placed at an angle of 180 degrees to the following unit, so that the solution pipelines stay short. This has been desired even though this arrangement has its own drawbacks such as difficult instrumentation, electrification and construction of service platforms.

Recently some solutions have been presented, with the aim of getting all the extraction steps to face the same direction. These are described for instance in conference publications “Alta 1996 Copper Hydrometallurgy Forum”, Oct. 14-15, 1996, Brisbane, Australia: Hopkins, W.: “Reverse Flow Mixer settlers” and ”Randol at Vancouver ’96”, Conference Proceedings, November 12-15, 1996, Vancouver, British Columbia, pages 301-306. In the latter publication there is a drawing on the bottom left of page 302 presenting a principle drawing of four different separation sections. The first is a conventional model, where the dispersion is fed from one end into the separation section and the separated solutions are removed from the other end. The next is known as the Krebs model, also described in US patent 4,844,801, which has the characteristic that the dispersion is conveyed along a launder overhead the settler to the end of the settler furthest from the mixer. There the dispersion is routed into the actual settler space to flow towards the mixer. The third is the Falconbridge model, where the settler is separated with partial partitions and the dispersion flows in the first half of the settler away from the mixing section and in the second half back towards the mixing section. According to the caption, the residence time of the solution in the settler depends on whether the solution is in the inner or outer edge of the settler. In the fourth, the Bateman model, which is also described in US patent 5,558,780, the dispersion flows along a narrow channel in the side of the settler to the furthest end of the settler and from there in the actual settler space back towards the extraction mixing section. The two latter represent what is known as reverse flow-type settlers.

In the Falconbridge model it is possible that the dispersion that has flowed along the inner edge of the settler does not have time to separate into its own phases so well as that which flowed along the outer edge. The principle drawing does not show in more detail how the flow is reversed in practice either. The settler described in US patent 5,558,780 has its own problems forming a uniform return flow in the settler. As a result the separation capacity of the settler remains incomplete and the entrainment of residual droplets in the separated solutions is high.

A method has now been developed in accordance with the present invention, where the dispersion formed in the mixing section of a metal extraction process is routed to a separation section, divided substantially into three parts with a partition wall in the same direction as the sidewalls. The dispersion and the phases separating from it flow first as an outward flow from the central part of the separation section to the rear end, where the entire amount of solution flowing in the separation section is reversed as two return flows towards the front end of the separation section. The return flows take place on either side of the outward flow. The outward flow of solutions is regulated to be dispersion-dominant i.e. the dispersion is kept as a strong layer in the outward flow side of the separation section by means of a reversing element situated in its rear end, because a strong dispersion band helps the formation of pure solution phases. In addition, the reversing element divides the separated solutions into sub-flows, which ease the reversal of the solution stream into return streams. In order to maintain a strong dispersion band the cross-section of the outward flow field also preferably decreases in size towards the rear end of the separation section and the cross-section of the return flow fields diminishes in size towards the front end of the separation section as well, too. The dispersion that has flowed through the reversing element and the separated solutions are conducted through a picket fence at the front end of the return flow fields, by which means the direction of the solutions is reversed finally towards the front end of the separation section.

The present invention also relates to a settler equipment, where an substantially rectangular settler is comprised of a front and rear end as well as sidewalls and a bottom. The width of the settler is substantially greater than its length. The settler is divided by partition walls into three sections, whereby the partitions extend preferably to a distance that is 85-95% of the total length of the settler. Three flow fields are formed in the settler by means of the partition walls, an outward flow field and a return flow field on either side. The settler partitions are positioned between the sidewalls substantially in the direction of the sidewalls, but nevertheless preferably in such a way that the cross-section of the outward flow field decreases towards the rear end of the settler and the cross-section of the return flow fields decreases towards the front end of the settler. At least one reversing element is positioned in the outward flow field in the immediate vicinity of the settler rear end, formed of an element extending from one partition to the other. The function of the reversing element is to regulate the thickness of the dispersion band and to achieve the controlled turning of the different phases in the rear of the settler. There are picket fences made between the rear end and the partition wall on the return flow field sides, which straighten out the settler flow towards the front end of the settler. The settler, which is equipped with two return flow fields, is particularly suitable for extraction applications, where the solution streams are large.

The substantial features of the invention will be made apparent in the attached claims.

The flow of the dispersion and separated phases from the front end of the separation space towards the rear end is called the outward flow and the flow of all these phases from the rear end of the separation space back towards the front end is called the return flow. Likewise the area of the settler where the outward flow occurs is called the outward flow field and correspondingly the fields on both sides are called return flow fields.

A dispersion from a liquid-liquid extraction mixing section is fed in the desired manner into the front end of the separation section in the outward flow field. Obviously, the aim is to spread the flow across the entire cross-section of the outward flow field. To further this picket fences or other suitable elements can be used. At least some of the equipment belonging to the mixing section such as mixers, which may number one or two, perhaps even three, may be placed either in front of the settler or inside the settler, at the front end of the outward flow field. For example US patent 5,185,081 describes an arrangement where the mixers are situated inside the settler. To prevent the dispersion that is discharged from the last mixer from flowing directly towards the rear section of the outward flow field, it is preferable to reverse the direction of flow of the dispersion first towards the side corners of the front end of the outward field and only from there to reverse the flow towards the rear end. The direction of the dispersion towards the rear end succeeds best when using picket fences, which are suitably profiled. A gentle zigzag-shaped picket fence when seen from above has proved to be the most appropriate solution. Collection channels for the separated phases can further be placed in the rear end of the outward flow field, which will circulate the solutions that have separated in the outward flow field to the mixing section pump tank within the same extraction step. Of course the outward flow field can also be equipped with only one solution collection channel depending on the circulation requirements. The channels may be for instance channels of the type described in US patent 6,083,400 or other equipment suitable for this purpose.

In the method according to the invention, the outward flow is regulated so that it is dispersion-dominant, i.e. the dispersion is kept as thick band between the phases. To bring this about, at least one reversing element is placed at the rear end of the outward flow field, which regulates the thickness of the dispersion layer and the progress of the dispersion. The phases that have separated from the dispersion are made to flow relatively freely, but the

unseparated dispersion is dammed up by means of at least one reversing element placed in the rear part of the outward flow field for this purpose.

The reversing element extends as far as the sidewalls of the separation section outward flow field i.e. from one end of the partition wall to the other. The arrangement according to the invention includes at least one reversing element situated in the rear end of the outward flow field of the settler (separation section). The reversing element extends as far as the sidewalls of the separation section outward flow field i.e. from one of the sidewalls to the end of the partition wall. The reversing element for its part comprises at least two plate-like parts or reverser plates, placed at different heights and substantially perpendicular to the longitudinal axis of the settler (in the direction of flow of the solutions). The direction of flow of the dispersion in the area formed between the reverser plates, in the reversing channel, is almost vertical, because the dispersion is made to flow above or below each reverser plate into the reversing channel. Changing the direction of flow substantially vertical improves the separation of the dispersion into pure solution layers above and below the dispersion. The reversing element can be positioned at different stages of extraction such as both in the actual extraction and also in any washing and stripping separation sections.

It is characteristic of the method and equipment that the dispersion stream is prevented from flowing forwards directly by arranging a reversing element in the rear end of the outward flow field extending above this field. Preferably the reversing element comprises at least two plate-like components, which are situated against the outward flow. In order for the dispersion to move past the reversing element, in the first stage it must be pressed against the first plate-like component of the reversing element and under it into the reversing channel, which is formed between the plate-like parts of the reversing element. From the reversing channel the dispersion surface is made to rise so that extends to flow over the second plate-like part of the reversing element. There are at least two plate-like parts in one reversing

element, but the number of said parts can also vary. The first plate-like part of the reversing element, or underflow plate, and subsequently every second part is located substantially higher in the separation section than the second plate-like part, or overflow plate, and every other part after that.

The first plate-like part belonging to the reversing element, the underflow plate, is located in the separation section at a height where its upper edge extends above the dispersion band into the organic solution phase. When the separated solutions and the dispersion band between them flow from the feed end of the separation section towards the rear end, the dispersion band is pressed against the first reverser plate. The dispersion should accumulate in such quantities that since being heavier than the separated organic solution, it penetrates from under the underflow plate, through the riser channel or channels between the reverser plates and from there on to the rear end of the separation section, where the dispersion and separated phases are turned back to the return flow field. The larger the settler, the larger the flow required. A dense dispersion attains an improved degree of solution separation, in other words, the amount of entrainment in each solution, both aqueous and organic, is decreased.

The first reverser plate, the underflow plate, is basically solid, but it is equipped with vertical slots or a slotted zone in its upper and lower section. The upper edge of the plate is whole and the slotted zone starts just below it. The upper edge of the plate and its slotted zone extend into the organic solution. The height of the slotted zone of the upper section of the plate is 5 – 25% of the total height of the reverser plate and 1 –10% of the total solution height in the rear of the separation space. The organic solution flows via the slotted zone into the rear of the settler divided into several sub-flows, in practice 10 – 100. Dividing the solution into sub-flows aids its smooth turning from the rear towards the return flow fields.

The lower edge of the underflow plate is whole, but immediately above it there are vertical slots. The height of the slotted zone is about 10 – 40 % of the total height of the plate. The lower edge of the underflow plate extends to the bottom part of the separation section. In practice the lower edge of the underflow plate is at a distance from the bottom equivalent to 15 - 30 % of the total height of the solution (solution depth) of the separation section (settler). The dispersion dammed up in front of the underflow plate flows via the slotted zone of the lower section into the riser or reversing channel between the reverser plates. The lower section of the slotted zone also helps to divide the aqueous solution flowing in the bottom at least partially into sub-flows, which promotes the smooth reversal of the aqueous solution in the rear section. The number of sub-flows is in practice the same as in the organic solution.

The second reverser plate, the overflow plate, of the reversing member is the same type as the first i.e. basically solid. The upper edge of the overflow plate is equipped with a slotted zone like that described above in relation to the upper edge of the underflow plate. The purpose of the slots in this case too is to promote the even distribution of the dispersion into the rear of the separation section. The lower edge of the overflow plate is placed clearly lower than the lower edge of the underflow plate, but in such a way however, that there remains unimpeded flow space for the separated aqueous solution. In practice, the lower edge of the overflow plate is at a distance from the bottom that is 3 – 10% of the total solution height in the separation section. The upper edge of the overflow plate is placed below the surface of the organic solution. In practice the upper edge of the second reverser plate is placed below the solution surface at a distance that is 20 – 40% of the solution height in the separation section. The distance between the underflow plate and the overflow plate is specified so that the rising speed of the dispersion in the reversing channel between the plates is in the region of 0.05 – 0.3 m/s. In practice this means that the distance between the plates is around 0.5 – 2 m, when the feed of the dispersion into the separation section

is over 1000 $m^3/h$. If the reversing element comprises several reverser plates, the slotted zones are placed in the upper and lower edges of the corresponding plates.

It is advisable to place flow blocking plates in front of the upper section of the overflow plate, which are made up of solid plates in the direction of the overflow plate. Blocking plates are placed in the slotted zone of the overflow plate. The height of the blocking plates can be changed. Blocking plates are set in the immediate vicinity of the overflow plate and by adjusting their vertical position the desired part of the slotted zone of the overflow plate can be covered. When the blocking plate covers the entire slotted zone, the surface of the dispersion band rises to the level of the upper edge of the overflow plate and blocking plate. When the upper edge of the blocking plate is lowered, the thickness of the dispersion band decreases and the thickness of the organic phase layer becomes thicker. In practice, the overflow plate blocking plate is comprised of several parts, each of which can be adjusted individually. Thus it is possible to balance the sideways flows of the entire outward flow field. The same function can be achieved by lifting or lowering the whole overflow plate, but in practice implementing this is more difficult, at least in large extraction facilities.

In most extraction applications the organic solution layer is not as thick as the aqueous layer. With the method and equipment according to the present invention it is possible to increase the area of the organic phase in the rear space of the separation section by positioning the reversing element plates to deviate from the vertical so that the plates are inclined against the outward flow. This means that the plates are placed at a 10 – 30° angle to the vertical, so that their lower edge is nearer than their upper edge to the rear end of the separation section. The purpose of inclining the reverser plates is to obtain a location of the dispersion band in the vertical direction at a level that corresponds to the final interface of the organic and aqueous phase in the return flow field. This furthers final phase separation in the return flow field.

The separated phases that have flowed via the reversing element into the rear of the settler and the dispersion band flowing between them are made to turn in the rear space back towards the front end of the settler by conducting them through picket fences of special construction. The picket fences reverse the return flow longitudinally towards the front end of the settler. The picket fences are supported at one end to the end of the partition wall and the other to the sidewall, either near the back wall or at the corner formed by the back wall and the sidewall.

The picket fences situated at the front of the return flow fields are made up of a normal picket fence, with guiding plates positioned behind its vertical slots. Guiding plates are set behind the vertical slots of the picket fence in relation to the direction of solution flow i.e. they are at the front of the settler. Guiding plates are turned behind the vertical slots so that the solution flow channel is narrower at the sidewall of the separation space and wider near the partition wall. This kind of solution reverses the flow of the solutions along the length of the settler. The picket fence solution presented is described in principle in US patent 6,132,615. In it the structures of the picket fence are positioned substantially vertically, but it is characteristic of this embodiment of the invention that the structures form an angle to the vertical corresponding to the outward flow field reverser plates. In this case this means that the plates of the picket fence are inclined with the upper edge towards the front end of the settler. The picket fences extend down to the bottom of the settler.

The area between the rear reversing element and the picket fences, the rear space, is dimensioned so that the flow rate of the streams there, of both the separated phases and the dispersion, is around 0.15 – 0.3 m/s. Just before the rear space, the controlled reverse of the solution flow direction is achieved by means of the reversing element located at the end of the outward flow field and the picket fences placed immediately at the front of the return flow field. The inclination of the reversing element and the picket

fences also even out the turning of the flow. Other separation-improving elements may also be located in the return flow fields.

At the front end of the return flow fields the pure solutions that have separated from the dispersion are removed from the settler, the organic solution as overflow into the organic solution headbox and the aqueous solution into its own headbox. The headbox is located outside the actual settler in front of the return flow fields. When the mixers in the mixing section are located in the corresponding position in front of the outward flow field, this constitutes a space-saving solution. When all the extraction steps can be placed in the same direction, the pipelines can be shorter.

The equipment according to the invention is described further by the attached drawings, where

Figure 1 shows an arrangement of an extraction step according to the invention as seen from above,

Figure 2A is a side view of a principle drawing of the reverser plates of the reversing element,

Figure 2B is a principle drawing of the reverser plates of the reversing element as seen from the rear end,

Figure 3A is another principle drawing of the reverser plates of the reversing element seen from the side,

Figure 3B is another principle drawing of the reverser plates of the reversing element as seen from the rear end, and

Figure 4 is another arrangement of an extraction stage as seen from above.

The extraction step according to Figure 1 comprises a mixing section 1 and a separation section or settler 2. The mixing section includes in this case a pump tank 3 and mixers 4 and 5. The aqueous solution and organic solution are conducted first to the pump tank and from there on to the first and second mixer. Obviously, the number of pump tanks and mixers may vary

according to the amount of solution to be fed. The pump tank is preferably that described in US patent 5,662,871 for example.

Settler 2 comprises the front end 6, rear end 7, sidewalls 8 and 9 and partition walls 10 and 11 in principle in the same direction as the sidewalls. The partitions, however, are preferably placed so that the cross-sectional area of the flow fields formed diminishes in the direction of flow. The partition walls may form a 5 – 15° angle with the longitudinal axis of the settler. The tapering angle of the outward flow field is preferably around 15 - 25 °. The dispersion of solutions from the last mixer is directed to the front end 6 of the settler in the outward flow field 12 (not shown in detail in the drawing). The outward flow field is equipped with picket fences or other suitable elements 13 and 14 to control the solution flow. At the rear end of the outward flow field there is a reversing element 16, which itself is composed of at least two reverser plates, an underflow plate 17 and an overflow plate 18. The rear section of the settler, the back 19 comprises the space left between the reversing element 15 and the picket fences 22 and 23 located at the front end of the return flow fields 20 and 21. In addition to the picket fences 22 and 23 at the front, the return flow fields can also be equipped with other desired elements for controlling the flow.

The cross-sectional area of the return flow fields also diminishes in the direction of flow towards the front end. The cross-section of the return flow fields may be the same or they may also differ from one another. In the embodiment of the invention, where three separate flow fields are formed in the settler, the ratio of the settler width to its length is around 2 – 5.

The headboxes of the separated solutions in the settler are preferably placed in front of the front end 6, on the side of the return flow fields 20 and 21. Thus the organic solution is recovered as an overflow from the organic solution headbox 24 via either one or several discharge units 25, from either just one of the edges or both. In the same way the aqueous solution is

recovered from the aqueous headbox 26 of each flow field via one or several discharge units 27 as required. The precise location of the discharge units is decided by where the separated solutions will be fed to. The headboxes may also be connected to each other appropriately.

Figures 2A and 2B present a decision in principle from the reversing elements situated in the back of the outward flow field. The drawings show that the underflow plate 17 and the overflow plate 18 are located near the rear end 7. The upper edge of the underflow plate is equipped with a slotted zone 28, which extends inside the separated layer of the organic phase 29. The slotted zone distributes the organic solution to flow into the back of the settler as several sub-streams. The underflow plate dams up the dispersion 30 flowing between the separated solutions, and the dispersion is made to rise through the slotted zone 31 in the lower edge of the underflow plate into the reversing channel 32, and from there through the slotted zone 33 in the upper part of the overflow plate into the back of the settler. The lower edge of the underflow plate is unbroken and is extended into the separated aqueous solution 34, but, however, above the bottom 35. The aqueous solution flows at least partially via the lower section of the slotted zone, so that it is made to divide into sub-flows and this aids the reversal of the solution flow direction in the back of the settler. The surface 36 of the organic solution phase is likewise the solution height of the settler.

Figures 3A and 3B show another embodiment of a reversing member, in which a solid blocking plate 37 is placed in front of the slotted zone 33 in the upper part of the overflow plate 18. The blocking plate is a plate that can be raised and lowered by means of its support structures 38 in the direction of the overflow plate. The height of the slotted zone 33 in the overflow plate of Figure 3B is far greater than that shown in Figure 2B, but it is now possible using the blocking plate to adjust the thickness of the dispersion and at the same time also that of the organic phase. In the case shown in the drawing the blocking plate is in the position whereby the lower part of the slotted zone

is covered by the blocking plate. In practice this means that the dispersion band is able to discharge into the back of the settler at the level of the upper edge of the blocking plate, so that the layer of organic phase can become thicker than for instance in the case of Figure 2. When the blocking plate is in its upper position, it can even cover the slotted zone completely and the dispersion band becomes thicker and the layer of organic phase becomes thinner.

Of course it is clear that the blocking plate can be installed to operate in other ways than that described above, but it is substantial that the thickness of the dispersion band and likewise that of the organic phase can be adjusted by closing part of the slotted zone of the overflow plate. As stated earlier, it is preferable to construct the blocking plates from several separate components, so that the layer thickness can be adjusted locally.

The embodiment of the extraction stage mixing section according to the invention shown in Figure 1 is located in front of the settler section, between the headboxes. The embodiment shown in Figure 4 is particularly applicable to large solution flows. In this case the mixing section 1 is equipped with two pump tanks 3, which are situated between the headboxes in the back of the return flow fields 20 and 21. The actual mixers are situated inside the outward flow field 12 of the settler 12. The organic solution and the aqueous solution are fed into the first mixer 4 from either one or both of the pump tanks. The mixed solutions are routed from the first mixer tangentially to the second mixer 5 via channel 39. The well-mixed dispersion is guided from the last mixer 5 to the settler space and there the dispersion stream is directed to discharge first towards the front end 6. In order that the dispersion stream does not flow between the mixers directly towards the rear of the outward flow field, the outer tracks of the mixers are connected to each other on the rear side with a partition wall 40 extending to the bottom of the settler. The direction of the dispersion stream is turned from the front corner 6 towards the rear end 7 of the settler. The front end of the outward flow field is prefer-

ably equipped with several picket fences 13, 14 and 15. The first picket fence 15 may be a conventional straight picket fence, and is in two sections so that the first section extends from mixer 4 to the nearer partition wall 10 and the second section of the picket fence extends from mixer 5 to the other partition wall 11. The following picket fences 13 and 14 preferably form a gentle zigzag shape when seen from above. There is at least one zigzag-shaped fence. The function of the picket fences is to direct the passage of the different phases and the dispersion directly towards the rear of the settler.

It has been also described in the rear end of the outward flow field how the internal flow of the extraction step can be implemented. Collection channels for the separated phases are located in the outward flow field before the reversing element 16, by means of which the some of the separated solutions can be recirculated. The drawing shows the aqueous solution channel 41 and the organic solution channel 42. According to the drawing the channel extends across the entire cross-section of the outward flow field. There is a pipeline 43 from the aqueous solution channel and a pipeline 44 from the organic solution channel to the same extraction step pump tank. The flow of the dispersion and the separated solutions to the reversing element and onwards from there occurs in the same way as that shown in Figure 1.

The method and equipment of the present invention now make it possible to handle even large solution streams economically and operationally in a cost-effective extraction step solution comprising a mixing section and reverse flow separation section as described above. Using the method and equipment of the present invention first of all the thickness of the dispersion band of the stream can be controlled and thus achieve pure solutions. Secondly, a controlled reversal of the outward flow field into return flow fields can be achieved by means of a regulating and reversing element in the rear section of the settler.